ELECTROLYTIC PREPARATION OF POTASSIUM CHLORIDE

BY

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ARMOUR INSTITUTE OF TECHNOLOGY

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WILLIAM K. MUNN AND FONTENELLE L. MOORE

TO THE

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THE ELECTROLYTIC PREPARATION OF POTASSIUM CHLORIDE: WITH TEST OF A NICKEL-CHROMIUM ELECTRODE.

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24131

We desire to acknowledge our indebtedness to Professor H. McCormack for his valuable suggestions offered during the work.

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PREFACE.

The electrolysis of the alkali chlorides forms one of the largest electrochemical industries which is carried out in aqueous solution. The hypochlorites, chlorates, and perchlorates of sodium and potassium, chlorine, and the hydrates aneobtained with varying conditions of electrolysis.

This thesis embodies a study of the behavoir of alkaline solutions of potassium chloride under electrolysis, with a view to its application to the commercial production of chlorate. A portion of the work is devoted to the test of a non-corrosive alloy of nickel, cobalt, and chromium as an electrode. A short discussion of the theory of the reactiobs and its development, and of the commercial applications is also given.

Chicago, May, 1913.

The Chemical Action of Chlorine on Water and Alkali Hydrate.

On electrolyzing a solution of potassium chloride, the first products obtained are chlorine at the anode, and potassium hydrate at the cathode. If these products are allowed to mix, the reactions take place that result in the formation of the hypochlorite and the chlorate.

Chlorine and water react together to a slight extent according to the following reaction;

$$Cl_2 + H_2O \rightleftarrows H^+ + CL^- + HOCl^- \qquad (1)$$

The equilibrium constant of this reaction is

$$K_1 = \frac{Cl_2}{H \cdot Cl \cdot HOCl} = 2570$$

Since a saturated solution of chlorine in water at 25° contains only 0.064 mols per liter, it is evident that only very small amounts of hydrochloric and hypochlorous

acids can exist simultaneously in solution the tendency of the reaction being from right to left, giving free Cl_2.

Chlorine reacts with alkali hydrate according to the following reaction;

$$Cl_2 + OH = HOCl + Cl. \qquad (2)$$

$$K_2 = \frac{Cl_2 \cdot OH}{Cl \cdot HOCl} = 3.6 \times 10^{-11}$$

Hypochlorous acid then reacts with the unchanged hydrate to form hypochlorite;

$$HOCl + OH \rightarrow OCl + H_2O \qquad (3)$$

$$K_3 = \frac{HOCl \cdot OH}{OCl} = 3.8 \times 10^{-7}.$$

The sum of equations (2) and (3) is usually taken to represent the reaction between chlorine and hydrate;

$$Cl_2 + 2\,KOH = KCl + KOCl + H^2O \qquad (4)$$

Since the equilibria for K_2 and K_3 exist simultaneously, the values of OH and OCl are the same in both.

$$\frac{HOCl}{OH} = K_3 \frac{OCl}{OH} \qquad (5)$$

and

$$\frac{Cl_2}{Cl} = K_2 \frac{HOCl}{OH} = K_2K_3 \frac{OCl}{\overline{OH}^2} \qquad (6)$$

This equation represents the equilibrium existing, and may be used to determine the effects of any changes in concentration.

Formation of the ClO_3 ion.

The formation of the ClO_3 ion takes place according to a number of reactions, according to conditions.

1. By direct oxidation.

$$Cl + 3O = ClO_3. \qquad (7)$$

$$ClO + 2O = ClO_3 \qquad (8)$$

$$3Cl_2 + 3O = 5\ Cl + ClO_3. \qquad (9)$$

2. By the action of chlorine on alkali hydrate.

$$Cl + 5OH = ClO_3 + H + H_2O \qquad (10)$$

3. By the action of hypochlorite on alkali hydrate.

$$ClO + 3OH = ClO_3 + H + H_2O \qquad (10)$$

4. The oxidation of hypochlorite by hypochlorous acid.

$$2HOCl + OCl = ClO_3 + 2Cl + 2H \qquad (11)$$

The free hydrochloric acid then sets free an equivalent amount of hypochlorous acid, and this in turn oxidizes more hypochlorite.

By a consideration of equation (6) we may trace the course of the reactions. When an amount of chlorine equivalent to the hydrate has been added, the solution is in stable equilibrium with a small amount of free hypochlorous acid present. An excess of chlorine, however, increases the concentration of the hypochlorous acid to such an extent that reaction (11) proceeds, and the hypochlorite is oxidized to chlorate.

The addition of a small amount of free acid would have the same effect in setting free hypochlorous acid.

Chlorate is produced in alkaline solutions by the reaction

$$3KOCl = KClO_3 + 2KCl. \qquad (13)$$

In weakly alkaline solutions the velocity of the reaction is small, but increaseson heating and with increase in alkalinity.

Electrolysis of Neutral Solutions of Alkali Chlorides.

When a concentrate neutral solution of alkali chloride is electrolyzed without a diaphragm, the metal is deposited on the cathode and reacts with water to form hydrate. On the anode chlorine is liberated as free chlorine. The chlorine and alkali hydrate react together as follows;

$$Cl_2 + 2KOH = KCl + KOCl + 2H_2O \quad (14)$$

The hypochlorite becomes more and more concentrated until a limiting value is reached. At the anode the hypochlorite ion is liberated, being more easily discharged than the chlorate ion, and chlorate is formed according to the reaction

$$6ClO + 3H_2O = 2ClO_3 + 4Cl + 6H + 1\tfrac{1}{2}O_2 \quad (15)$$

This is called the anode chlorate formation. At the cathode the reduction of hypochlorite takes place according to the following;

$$KOCL + H_2 = H_2O + KCl \quad (16)$$

This reduction is nearly entirely prevented by the addition of a small amount of potassium chromate to the solution. The action consists in the formation of a diaphragm of oxide of chromium on the cathode.

Electrolysis in Alkaline Solutions.

Oettel(1)found in his experiments that as the alkalinity of the solution was increased , the maximum concentration of the hypochlorite became less and the principal product of the electrolysis became chlorate. The accompanying curves(2) show the effect of increasing alkalinity on solutions of sodium chloride of 20% strength.

The increase in chlorate formation is explained as follows:

The reaction takes place in the same manner as in neutral solutions, except that the chlorine ions find hydroxyl ions with which to react immediatley. There is therefore a higher immediate concentration of hypochlorite, and consequently its discharge and the production of

(1) Z.F. Elektrochem. 1-474.

(2) Muller, Z.F. Elektrochem. 6-20.

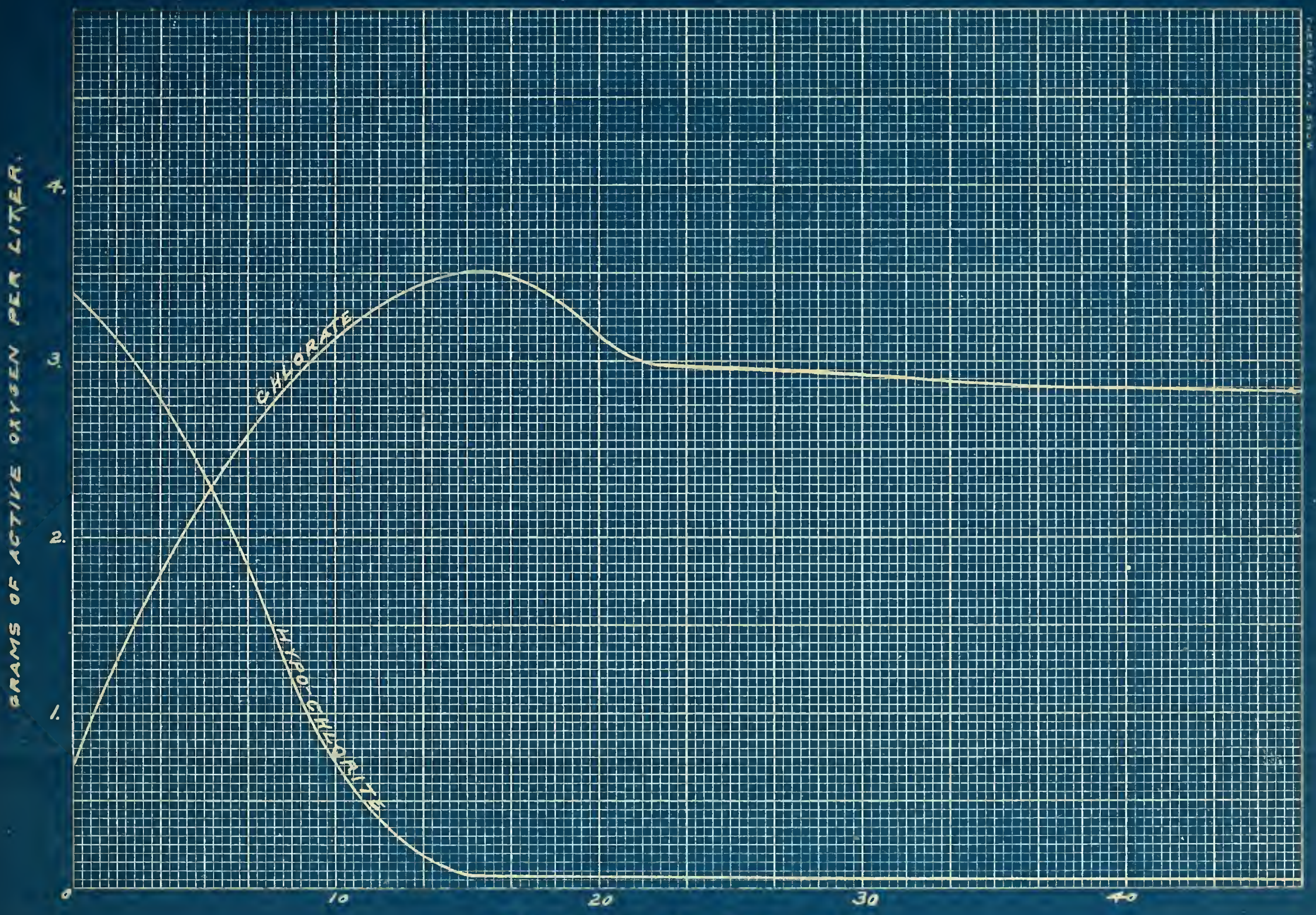
GRAMS OF ACTIVE OXYGEN PER LITER.
4.
3.
2.
1.
0
10
20
30
40
GRAMS OF NaOH PER LITER
CHLORATE
HYPO-CHLORITE
FREEMAN AND SNOW

Fig. 1.

chlorate takes place when the concentration throughout the solution is very low. When the alkalinity is further increased, the discharge of hydroxyl ions takes place and the yield falls below a maximum as shown by the chlorate curve.

The account of the tests given in part IV . illustrates another striking difference between the behavoir of neutral and of alkaline solutions. In the former the solution has to be heated to about 60° before the concentration of the chlorate becomes large. In strongly alkaline solutions the temperature effect is just the reverse, as is shown by the curves in part IV.

Foerster(1) and Muller attempt to explain this effect of the temperature, but it seems that the action is not very

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(1) Foerster, Elektroch. Wasseriger Losungen, pp. 366-369.

understood. It is evident, however, from a study of equation (6) that with increase in temperature there is an increase in concentration of the free chlorine, and consequently an increase in the concentration of the OCl ion. In neutral solution this is oxidized by the free hypochlorous acid to chlorate, according toequation (12). In strongly alkaline solutions, however, the increased concentration of the OH decreases the concentration of the HOCl to a very small quantity, and the formation of chlorate tak takes place only by the discharge of the ClO ions, according to equation (14). Consequently, the only effect of the increase in concentration of the free chlorine is an increase in hypochlorite concentration.

Summary.

A brief account of the important developments in the theory and experimental work on the electrolysis of the alkali chlorides is of interest at this point. The reactions between chlorine and alkali hydrate were noted soon after the discovery of the element, but did not receive much study until recent years.

The first experiments of importance were those of Hausermann and Naschold, in 1894. The attributed the formation of chlorate to primary reaction. Oettel, in his experiments of about the same date, advanced the conclusion that the formation of chlorate was due in neutral solutions to secondary reaction, and to primary reaction in alkaline solutions.

The use of gas analysis as a means of controlling the cell, consisting in the comparison of the evolved gases with those

from a water coulometer in series with the cell, was first devised by Oettel.(1)

Another investigator, Wohlwill, working about this time, states that the formation of hypochlorite occurs as a reaction between Cl and OH at voltages of over 2.2. Chlorate formation he attributes to the reaction

$$2ClO \quad OH \quad HClO_3 \quad Cl_2$$

The most exhaustive work on the subject is that of Feerster and Muller. They attributed the formation of chlorate to secondary reactions in both neutral and acid solutions, substantially according to the theory given in the preceding pages. The action of potassium chromate in the prevention of cathode reduction was worked out by these investigators.

Some recent work by Betts(2) and

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(1) Pract. Exercises in Electrochem. pp.43-46.

(2) Jour. Am. Chem. Soc. 29-340.

Sherry brings out some interesting results with reference to the cathode reduction. Additions of salts of tungsten, thorium, cerium vanadium, and some others were tried and have to a certain extent the same action as the chromates. A cathode of metallic magnesium gives about the same efficiency as the addition of chromate, due to the formation of a diaphragm of magnesium oxide or hydroxide on the cathode. The metal is however, corroded quite rapidly, about in the proportion of one part of magmesium per fifty parts of chlorate made.

Technical Processes for the Manufacture of Chlorate.

The earliest process worked out to a technical application was that of Watt, in 1851. The process used a warm, saturated solution of potassium chloride, which flowed between electrodes and out into crystallizing tanks, where it was cooled and the chlorate removed. No record of the commercial application of the process is given.

The process of Gall and Montlaur, patented in 1884, has been used to a considerable extent, being the first process to receive a wide application. The cell originally contained a diaphragm to prevent reduction, the solution circulating by means of external pipes. Later this was given up, the addition of chromate being used to prevent reduction. The anodes were of

platimum and the cathodes of iron, or better, nickel. In the early process, concentrated solutions and high current density were used, and the chlorate was allowed to crystallize out in the cell, and was raked out with iron rakes. The later process is generally modified so that the solution is withdrawn to the crystallizing vats before it becomes saturated with chlorate. The mother liquors from the crystallizations are made up to strength with fresh chloride and returned to the cell. Plants operating this process are at Vallorbe, and St. Michael, in the Savoy.

The National Electrolytic Co., at Niagra Falls, makes potassium chlorate under the patents of W.T. Gibbs(1).

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(1) U.S. Pat. #665426-665427-665679.
(1901)

A brief description of the process is given by Richards(2). The anodes are of platinum and the cathodes of copper or iron, placed about three mm. apart. The cell is a frame 18" x 26" and any number of these may be clamped together like a filter press. The solution of potassium chloride flows in continuously and is heated to 70° by the action of the current. After coming from the cell, the solution is cooled in crystallizing tanks, the crystals removed, brought up to strength with chloride, and returned to the cell. An efficiency of 70% is claimed.

A recent plant uses a cell with platinum anodes and brass cathodes. The electrolyte is an acid solution of pot-

(2) Electrochem. Indust. 1-19 (1902).

assium chloride, containing bichromate. The solution flows in, is heated to 60-70° by the current, is drawn off to crystallizing tanks and crystallized in the usual way. Figures on the efficiency are not given out, but will approach 90% of the theoretical.

The crystallization apparatus is an important part of the chlorate plant, and the arrangements for expeditious and economicalmhandling of the sloutions require the careful consideration of the chemical engineer. Most of the chlorate made is purified by recrystallization, hence the success or failure of a process may depend on the efficiency of this section.

Experimental Work on the Electrolysis of Potassium Chloride Solutions. Test of the Nickel-Cobalt-Chromium Anode.

The electrolysis of chloride solutions belongs in the class designated as insoluble anode electrolyses. Also the conditions, namely the presence of an extremely active element, chlorine, at this point, renders it very difficult to find any substance which will fulfill the condition of insolubility. Platinum is of course the most acceptable material, but the large surface required and the consequent heavy investment is a serious drawback. A cell producing 100 lbs. of chlorate per day requires an anode area of 800 to 1000 square decimeters.

It was thought that one of the so-called non-corrosive alloys, which are coming to replace platinum for many uses,

namely an alloy of nickel, cobalt, and chromium, would fulfill the requirements as a substitute in this case.

The anodes were plates of the alloy about 2 x 3½ in. square and 1/16 in. thick. They were first ground to a smooth surface and polished, in order to reduce as much as possible the surface exposed to action. They were then tested in several instances under varying conditions.

In neutral solutions at current densities of ½ to 1 ampere per square decimeter, only a slight corrosion was noted. after one hours running. When the current density was increased to much above this, however, the action became very much more rapid. Plates run at 10 amp. per decimeter for an hour were found covered with a deep coat of oxides of the metals, and in some places corroded almost through

the plate. The solution was also contaminated with chlorides of the metals.

In other solutions the behavoir was practically the same. In alkaline solutions the corrosion was apparently a little more rapid. The results show that even at much lower currentdensities than are practicable for the work, a noticeable corrosion takes place. Another very undesirable feature would be the contamination of the solution with the dissolved anode material.

A few experiments were made in which the alloy was employed as a cathode. In this place it shows no particular difference from the other common cathodes.

Experiments on the Electrolysis of Alkaline Solutions.

The electrolysis of the alkali chlorides has been the subject of considerable experimental investigation of recent years, especially from the theoretical standpoint, and the development of the theory represents one of the best examples of the application of physico-chemical principles to a reaction. In regard to the technical applications. the work has been more limited, and of a more private nature, very little having been published in regard to the operation of any of the existing processes.

From a review of the experimental work, it will be noted that the electrolysis has been carried out mainly in neutral or acid solutions, Also

the technical processes all employ an acid solution.

The fact that in strongly alkaline solutions the principal product of the electrolysis becomes chlorate might lead to the supposition that such a solution would be suitable as an electrolyte for the production of chlorate. With this in view, we present the following series of experiments on the behavoir of these solutions.

Referring to Fig. 1,(p.8a) we note that as the alkalinity increases, the concentration of the chlorate reaches a maximum, thenfalls somewhat. It is of course desirable to select at once that alkalinity which gives the highest efficiency. This corresponds very closely to a solution containing 10% in KOH of the weight of KCl present.

The solution used except where otherwise noted contained 200gm. potassium chloride and 20 gm. potassium hydrate per liter.

The electrolyses were made in a glass cell holding 250 cc. of the solution. The anode was a platinum plate 0.87 sq. dm. in area. The cathode was of copper with about 2/3 of this area. The solution was stirred by a stream of air led into the cell. An ammeter and rheostat placed in the external circuit furnished the means of regulating and measuring the current. The whole cell was enclosed in an iron vessel, which could be packed with ice or filled with water and heated to any desired temperature.

The first runs were made using a current density ~~of~~ 5 amperes per sq.

decimeter at the anode. 250 cc. of the solution were placed in the cell and the current started. Readings of the ammeter taken at regular intervals and averaged gave the current passong. The electrolysis was continued for one hour, after which the solution was made up to a definite volume and an aliquot analyzed for hypochlorite and chlorate.

By suitabe regulation of the temperature, runs were made at this current density at approximatley 10°, 30°, 50°, 70o, and 90° C. The results are expressed graphically in Fig. 2, and tabulated in the following;

Result of Electrolyzing for One Hour a Solution Containing 200 gm. KCl and 20 gm. KOH per Liter

$D_A = 5$ amp./dm^2.

Temp.	Current	$KClO_3$	KOCl	% Yield.
5.8°	4.20	2.213	.126	70.8
33.0	4.29	2.538	.143	79.3
51.3	4.24	1.985	.154	62.6
75.2	4.25	2.110	.169	66.6
83.3	4.22	1.842	.169	58.6

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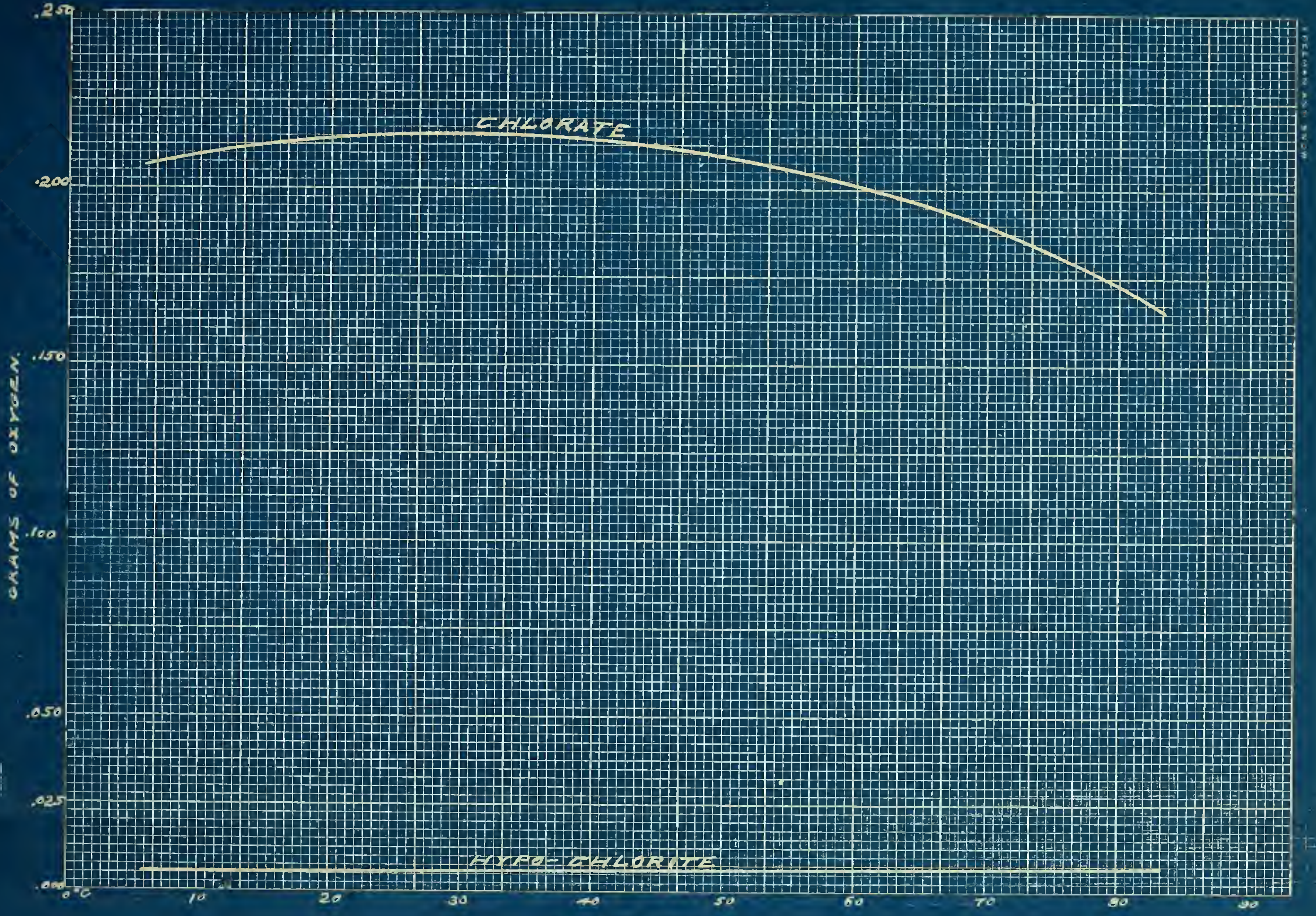
CHLORATE
HYPO-CHLORITE
.250
.200
.150
.100
.050
.025
.000
GRAMS OF OXYGEN.
0°C
10
20
30
40
50
60
70
80
90
TEMPERATURE.

Fig. 2.

The same runs were also made for current densities of 10 amperes and 15 amperes per sq. decimeter, with approximatley the same temperature regulation. The results are given in the following tables, and expressed in the curves, figs. 3 and 4.

The efficiencies given in the tables are based on the amount of chlorate formed, and do not take into account the amount of hypochlorite in the solution. The show, however, that the true current efficiency (hypochlorite + chlorate) diminishes markedly with increase in temperature. The hypochlorite concentration is at all times small, but shows a well marked increase as the temperature rises. The chlorate concentration diminishes with increase in temperature, as brought out in the theory, The yields ranging from 50% to 75%. The higher value is

Result of Electrolyzing for One Hour at 10 amp. per $dm.^2$

Temp.	Current.	$KClO_3$	KOCl	% Yield.
18.7°	9.99 A.	4.62 g.	.148g.	62.0
37.2	9.84	4.58	.169	62.4
52.1	10.12	4.17	.211	56.3
69.8	9.97	3.94	.230	52.8
85.3	9.42	3.91	.253	55.7

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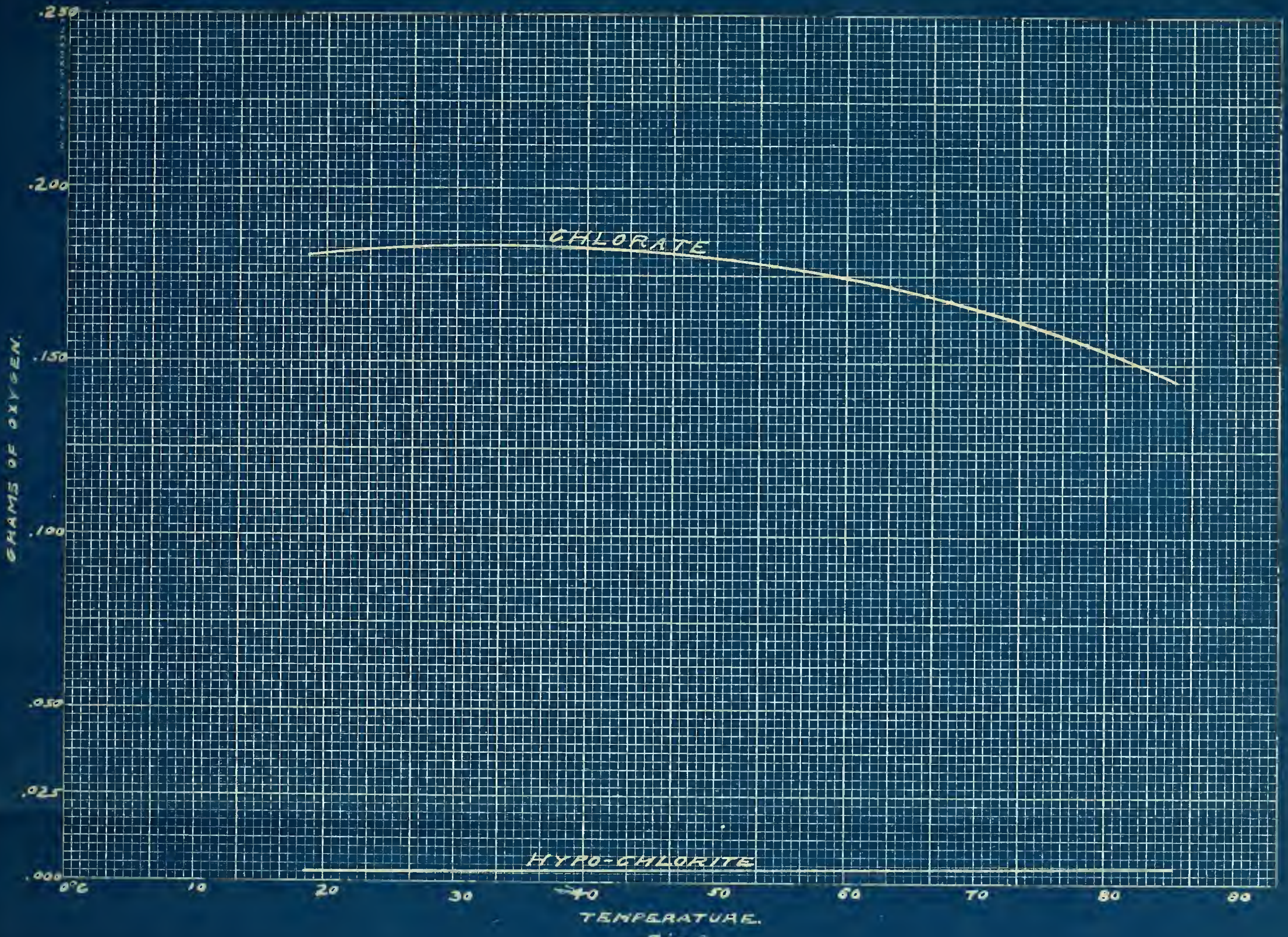
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.200
.150
.100
.050
.025
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GRAMS OF OXYGEN.
0°C
10
20
30
40
50
60
70
80
90
TEMPERATURE.
CHLORATE
HYPO-CHLORITE

Fig. 3.

Result of Electrolyzing for One Hour at 15 amp. per dm2.

Temp.	Current.	$KCLO_3$	KOCl	% Yield.
22.8	15.00 A.	6.90g.	.084	61.7
40.4	14.66	6.87	.145	62.8
54.4	14.68	5.95	.168	54.4
73.7	15.06	5.58	.211	49.8
87.0	15.00	5.42	.295	48.4

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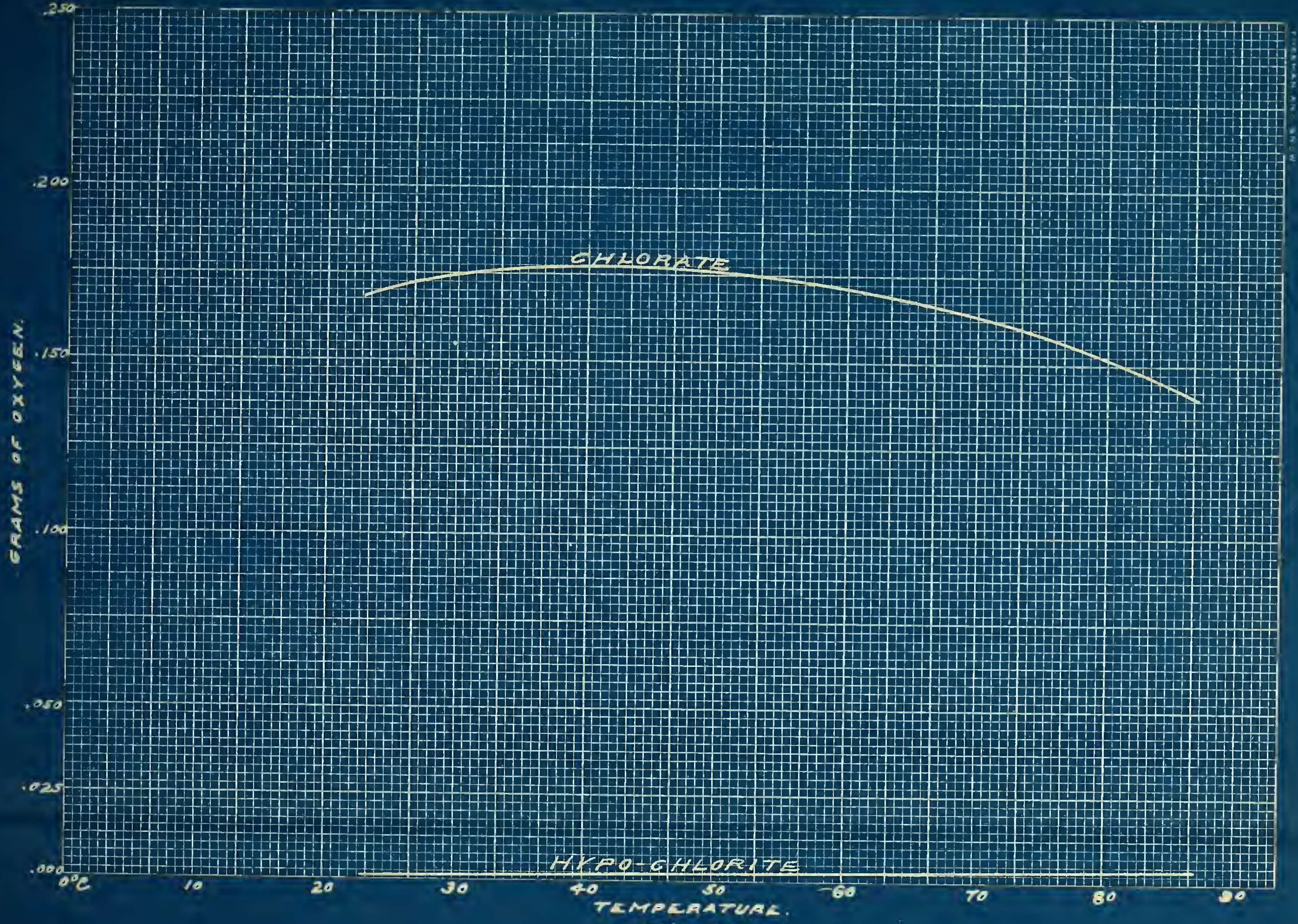
CHLORATE
HYPO-CHLORITE
GRAMS OF OXYGEN.
.250
.200
.150
.100
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10
20
30
40
50
60
70
80
90
TEMPERATUAL.

Fig. 4.

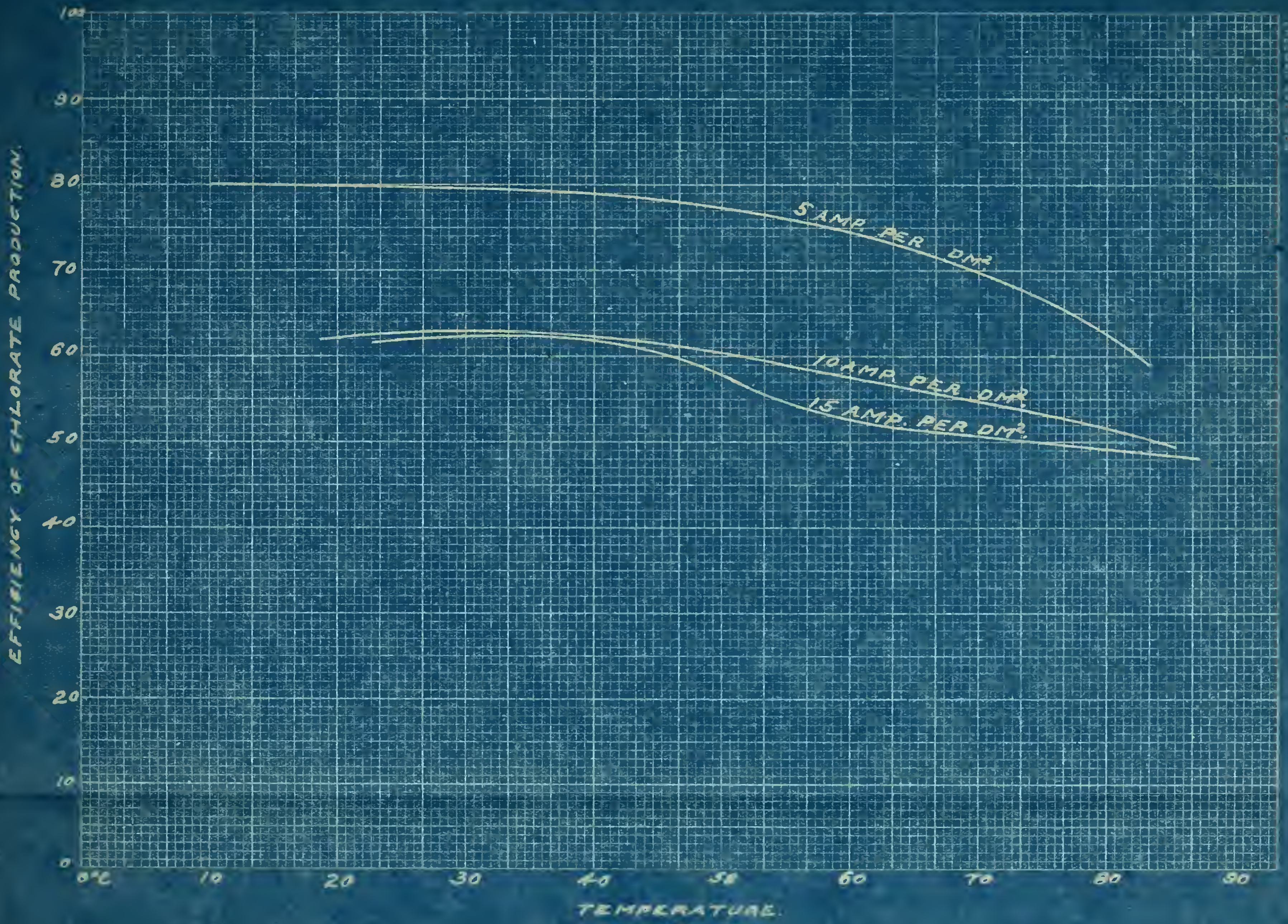
100
90
80
70
60
50
40
30
20
10
0
0°C
10
20
30
40
50
60
70
80
90
EFFICIENCY OF CHLORATE PRODUCTION.
TEMPERATURE.
5 AMP. PER DM².
10 AMP. PER DM².
15 AMP. PER DM².

Fig. 5.

obtained as usual with the smaller current density.

Fig. 5 shows the variation of the efficeincy with the temperature; the curves also serve to illustrate, from the relative position of the curves, the decrease in efficiency with increase in current density.

The addition of chromates to alkaline solutions increases somewhat the efficiency obtained, but in a less degree, since the cathode reduction is less in alkaline solutions. This is because the limiting concentration of hypochlorite is much lower than in neutral solutions. The following electrolyses illustrate the effect of the addition of 2 gm. potassium chromate to 250 cc. of the electrolyte.

Electrolyte Containing 2 gm. $K_2Cr_2O_7$.

Current	$KClO_3$	% Yield
10 amp. hr.	4.78 gm.	64,3
10 " "	4.89 "	65.8

* * *

Operation of a Commercial Cell.

In order to make a commercial test of the process, a cell was constructed in which the operation could be carried out on an industrial scale. The general construction is shown in figures 6 and 7. The container wasa glass jar 10" x 4" x 5#. Five platinum plates, each about one sq. dm. in area, served as anodes, these alternating with four cathodes of copper. The electrodes were placed about ½ cm. apart, being hung from two copper bus-bars of 100 amperes capacity. By placing the electrodes as close together as possible, the heating effect of the current is reduced.

The electrolyte consisted of a saturated solution of potassium Chloride,

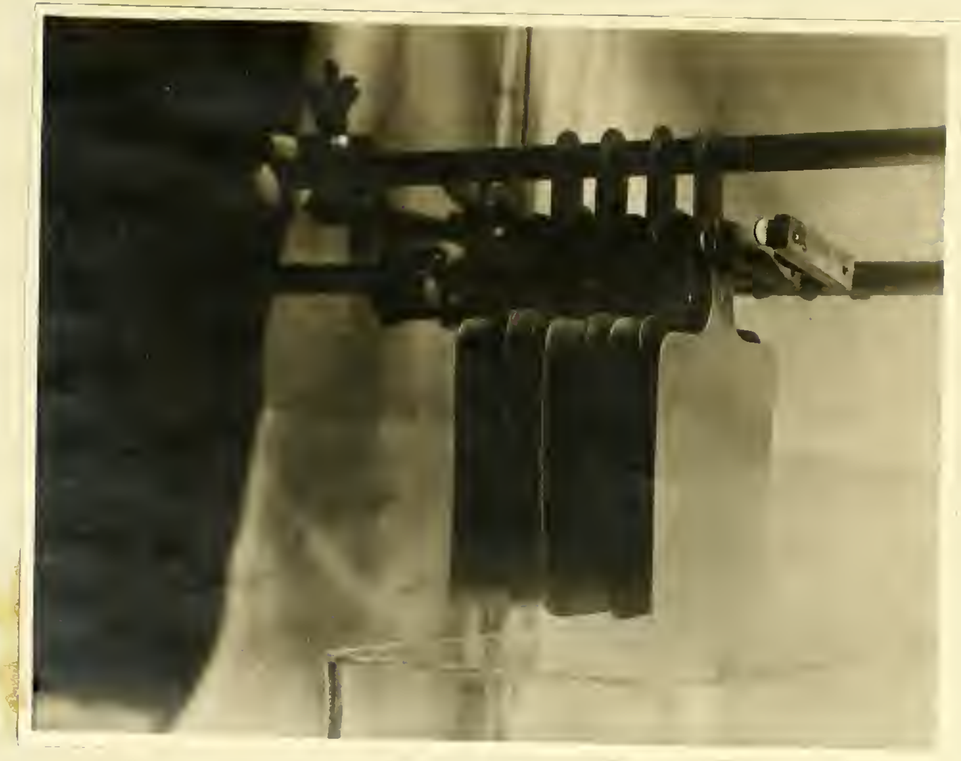

Fig. 6

with 10% of potassium hydrate, based on the weight of chloride, and two grams potassium chromate per liter. The electrolysis was made using a current density of 20 amperes per sq. decimeter on the anode. The total current input was 100 amperes. The circulation of the electrolyte was maintained at a sufficient rapidity so that the temperature did not rise above 30° C. Stirring was accomplished by means of a motor driven propeller.

Owing to the low solubility of chlorate,,the solution soon became saturated and chlorate crystallized out and was removed.

This cell will produce about three pounds of potassium chlorate per 24 hours.

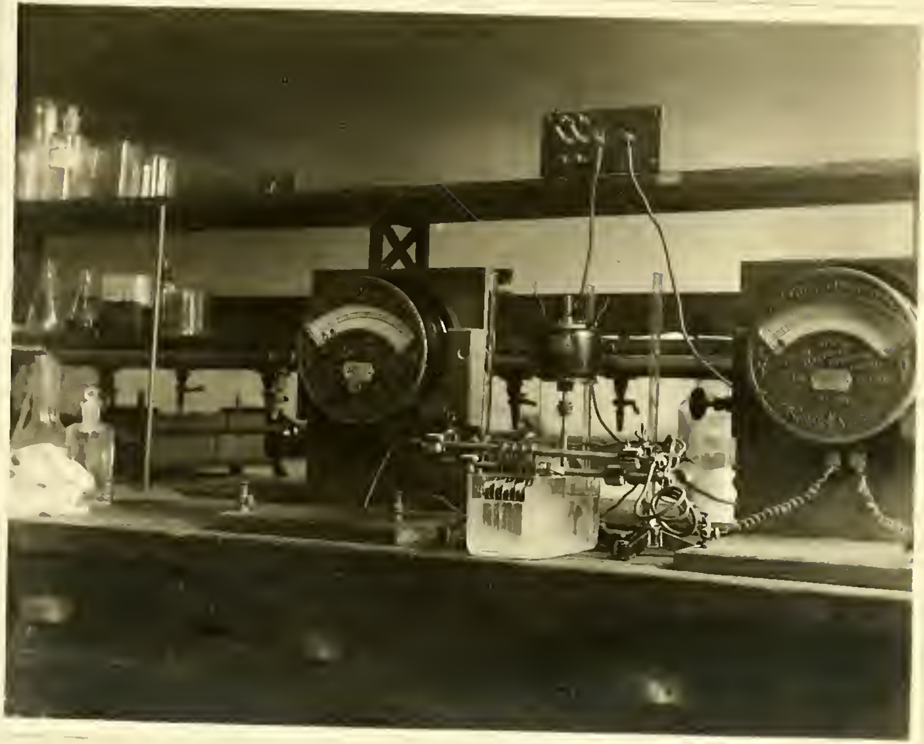

Fig. 7.

Methods for the Analysis of Solutions Containing Hypochlorites and Chlorates.

As a rapid and satisfactory method for the analysis of solutions containing hypochlorites and chlorated, the following was devised by the authors. The determination is based on the following reactions;

$$HOCl + 2KI = KCl + KOH + I_2$$

$$KClO_3 + 6HCl = KCl + 3H_2O + 3Cl_2.$$

The latter reaction takes place slowly in cold solutions, rapidly at boiling temperatures.

1. A measured volume of the solution is taken, acidified with hydrochloric acid, an excess of 10% potassium iodide added,

and the liberated iodine titrated with N/10 thiosulphate solution, a few drops of starch being added toward the end of the reaction.

2. Another portion of the solution is run into the decomposition flask of a Bunsen distillation apparatus, an excess of hydrochloric acid added, and t the chlorine distilled off into potassium iodide in the usual way. The liberated iodine is titrated with thiosulphate. The amount used corresponds to the sum of the iodine liberated by the chlorate and that liberated by the hypochlorite. Deducting from this the amount used by the hypochlorite in (1), we obtain the amount used by the chlorate.

1000 cc. N/10 thiosulphate = 1/10 I

$$\frac{KClO_3}{60} = 2.043 \text{ gm. } KClO_3.$$

1000cc. N/10 thoosulphate =

$$\frac{KOCL}{20} = 4.528 \text{ gm. } KOCl.$$

Owing to the fact that potassium chlorate is decomposed slowly in the cold by hydrochloric acid, the above method gives too high results for hypochlorite unless carefully carried out. The experience of the authors has been that if the titration is performed rapidly in cold solution, avoiding too large an excess of acid, that good results are obtained. The analyses of solutions in the preceding tests were made by this method, with consistent results.

Hypochlorite in the presence of chlorate may be determined by titration with a solution of arsenious acid(1).

(1) Treadwell and Hall
Quantitative analysis, p. 645.

$2KOCl + As_2O_3 = As_2O_5 + 2KCl.$

1000 cc. N/10 As_2O_3 = 3.546 gm. Cl.

The end point is reached when a drop of the solution ceases to give color on iodo-starch paper. This method is claimed to be more reliable than the iodimetric method, as the presence of chlorate has no effect in this case, but was not found as satisfactory as the former.

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